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(64) Gas separation

(67) A pressure swing adsorption (PSA) process for the separation of a component from a gas mixture also containing water vapour, eg the separation of nitrogen from air, employs adsorbers 1 and 2 each having a first stage 3, 3' containing a desiccant adsorbent and a second stage 4, 4' containing an adsorbent which preferentially adsorbs another component of the mixture eg a carbon molecular sieve which adsorbs oxygen more rapidly than nitrogen. Each adsorber is put through a cycle comprising an adsorption step during which the mixture is passed through the adsorber by a compressor 5 and unadsorbed product is withdrawn through a delivery line 11, and a desorption step during which the adsorber is vented to atmosphere through a waste gas

line 9. The pressure reached in the adsorber during such venting is the lowest reached during the entire cycle, no vacuum pump being employed. A valved line 12 allows pressure-equalisation between the adsorbers.

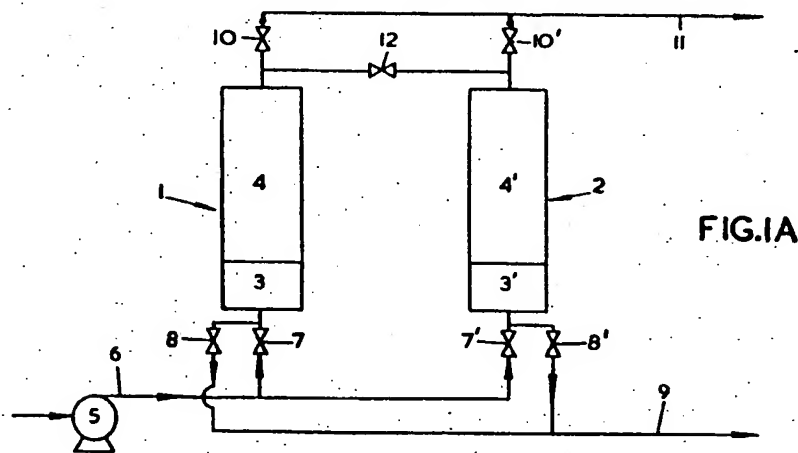


FIG. 1A

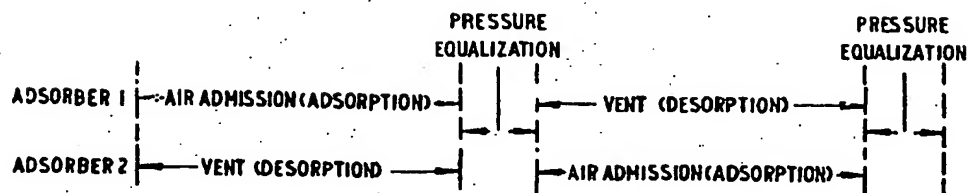


FIG. 1B

## SPECIFICATION

### Gas separation

5 The present invention relates to a method of gas separation by pressure swing adsorption (PSA) techniques, and is particularly, though not exclusively, concerned with a PSA process for the separation of nitrogen from air.

10 One known PSA process for the separation of nitrogen from air employs a molecular sieve carbon adsorbent which has the ability to effect a separation as between the two major components of air by virtue of its more rapid adsorption of oxygen than of nitrogen. In  
15 operation a bed of this adsorbent is put through a cycle which includes an adsorption step during which time air is pumped through the bed, most of the oxygen and a proportion of the nitrogen plus substantially all of the  
20 carbon dioxide and water vapour in the feedstock are adsorbed, and a nitrogen rich product gas is supplied from the outlet of the bed; and a desorption step during which time the outlet of the bed is closed, the bed is vented to  
25 atmospheric pressure through its inlet and then evacuated through its inlet, so that the adsorbed gases are substantially removed from the bed thereby preparing it for the next adsorption step. In practice to adsorbent beds  
30 are employed and operated on similar cycles but sequenced to be out of phase with one another by 180°, so that when one bed is on its adsorption step the other bed is on its  
35 desorption step, and vice versa. Furthermore it is usual to equalise the pressures in the two beds between each step by connecting the two bed inlets together and connecting the two bed outlets together; with these connections  
40 made the gas within the void spaces of the bed which has just completed its adsorption step is sucked into the bed which has just completed its desorption step by virtue of the pressure difference which exists between the  
45 beds at that stage, and this is found to be beneficial in maximising the product output because such void space gas will have already become somewhat enriched in nitrogen.

To effect the feed of air through the adsorbent beds a plant operating this process must  
50 include a compressor, and to evacuate the beds during desorption a vacuum pump is also included. Typically the bed pressure during adsorption rises to a maximum value of 7  
55 to 10 bar g (although lower adsorption pressures are sometimes employed), with the vacuum reached during desorption typically being in the region of 100 Torr. Plants operating the process as described above can readily  
60 achieve a nitrogen product purity in excess of 99% by volume.

Now, in order to reduce the capital and running costs of such a plant it has been  
65 proposed to eliminate the vacuum pump, so that during desorption the beds are vented to

atmospheric pressure but are not reduced to a pressure below atmospheric. Clearly if costs are reduced in this manner a penalty will be paid in that, for the same product purity and  
70 all other factors being equal, the plant will not be able to process the same volume of feedstock as a plant operating with a vacuum pump and thereby providing a greater overall pressure swing. Nevertheless, if the after-mentioned problem can be overcome such operation  
75 may make economic sense in many instances, and the operator of a plant initially assembled without a vacuum pump is given the flexibility to up-rate the performance of his  
80 plant at a later date if circumstances should so require, simply by installing a vacuum pump.

The problem which has been found in operating the process without a vacuum pump is that, over a period, a gradual decrease in the  
85 product output at a given purity is observed, in one experiment the product flow rate falling by some 40% over a period of six hundred hours. From a consideration of the water adsorption isotherm of the molecular sieve  
90 carbon adsorbent we postulate that the reason for this decreasing performance is that, without vacuum, a proportion of the water vapour adsorbed during the adsorption step remains on the adsorbent after the desorption step, so  
95 that as the cycle is repeated moisture gradually builds up in the adsorbent bed thereby decreasing its capacity to effect the desired oxygen/nitrogen separation.

It is an aim of the invention to overcome  
100 this problem and, while the invention is principally concerned with the separation of nitrogen from air using a molecular sieve carbon adsorbent the invention may also be applicable to other PSA separations where similar  
105 problems arise. As an example there can be quoted the separation of hydrogen from a mixture with methane using the same carbon adsorbent and where moisture is also present in the feedstock.

In a broad aspect the invention accordingly  
110 resides in a process for the separation of a desired gaseous component from a gaseous mixture including the desired component, at least one other component and water vapour,  
115 which process comprises repeating a cycle of operation including successive adsorption and desorption steps, the adsorption step comprising passing the gaseous mixture at a superatmospheric pressure through an adsorber having  
120 a first stage containing a desiccant adsorbent and a second stage containing an adsorbent which adsorbs said other component more readily than said desired component, so as to obtain from the adsorber a product gas  
125 enriched in said desired component and depleted in said other component and water vapour; and the desorption step comprising venting the adsorber to a pressure not substantially below atmospheric pressure in a  
130 direction counter to the direction of passing

to effect a nitrogen/oxygen separation in the same way as the beds of a plant operating the known process previously described, but that no vacuum pump is included in the plant

- 5 thereby reducing both its capital and running costs. Furthermore, the incorporation of desiccant beds 3 and 3' which effectively function as heatless driers upstream of the main carbon beds ensures that there can be no undesirable build up of moisture in the adsorbers.

- 10 Turning now to Fig. 2A this shows a modification of the Fig. 1A plant to provide an additional purging operation during the desorption steps of each adsorber. To this end a purging line 13 including a throttle valve 13A branches from the product delivery line 11 and in turn branches into two lines leading to the outlet ends of the adsorbers 1 and 2 controlled respectively by valves 14 and 14'.
- 20 At a predetermined interval after the opening of the valve 8 or 8' during the desorption step of the appropriate adsorber the valve 14 or 14' is also opened and remains open with valve 8 or 8' until the end of the desorption step. During this time a proportion of the product gas from line 11 is diverted through line 13 (the proportion being determined by the setting of throttle valve 13A) and flows downwardly through the appropriate adsorber thereby purging out the void space gas from each of the adsorber stages and replacing such gas with product-quality gas in preparation for the following adsorption step. Such a purging step may be especially valuable in an embodiment having the same adsorbents as described above but used for separating hydrogen from a mixture with methane and water vapour.

- 40 As a further variation of either plant indicated in broken line in Fig. 2A an additional line controlled by a valve 15 may be provided whereby the inlet ends of the adsorbers can be connected together during the pressure equalization steps.

#### 45 CLAIMS

1. A process for the separation of a desired gaseous component from a gaseous mixture including the desired component, at least one other component and water vapour, which process comprises repeating a cycle of operation including successive adsorption and desorption steps, the adsorption step comprising passing the gaseous mixture at a superatmospheric pressure through an adsorber having a first stage containing a desiccant adsorbent and a second stage containing an adsorbent which adsorbs said other component more readily than said desired component, so as to obtain from the adsorber a product gas enriched in said desired component and depleted in said other component and water vapour; and the desorption step comprising venting the adsorber to a pressure not substantially below atmospheric pressure in a

direction counter to the direction of passing the gaseous mixture, such pressure being the minimum pressure obtained in the adsorber during the cycle.

- 70 2. A process for the separation of nitrogen from a gaseous mixture including nitrogen, oxygen and water vapour, which process comprises repeating a cycle of operation including successive adsorption and desorption steps, the adsorption step comprising passing the gaseous mixture at a superatmospheric pressure through an adsorber having a first stage containing a desiccant adsorbent and a second stage containing a molecular sieve carbon adsorbent which adsorbs oxygen more rapidly than nitrogen, so as to obtain from the adsorber a product gas enriched in nitrogen and depleted in oxygen and water vapour; and the desorption step comprising venting the adsorber to a pressure not substantially below atmospheric pressure in a direction counter to the direction of passing the gaseous mixture, such pressure being the minimum pressure obtained in the adsorber during the cycle.

- 85 3. A process according to claim 1 or claim 2 wherein said desiccant adsorbent is alumina or silica gel.

4. A process according to any preceding claim wherein the desorption step includes, subsequent to venting the adsorber, purging the adsorber with a stream of said product gas in a direction counter to the direction of passing the gaseous mixture.

5. A process according to any preceding claim wherein the maximum pressure reached within the adsorber as a result of the adsorption step is not above 10 bar g

6. A process according to any preceding claim employing two such adsorbers and wherein each undergoes a similar such cycle of operation but sequenced to be out of phase with one another by 180° so that when one adsorber is on its adsorption step the other adsorber is on its desorption step, and vice versa.

7. A process according to claim 6 which includes the step of equalizing the pressures within the two adsorbers between each adsorption and desorption step.

8. A process according to claim 7 wherein said pressure equalization is achieved by connecting together the outlets of the adsorbers.

9. A process according to claim 7 wherein said pressure equalization is achieved by connecting together the outlets of the adsorbers and connecting together the inlets of the adsorbers.

10. A process for the separation of a desired gaseous component from a gaseous mixture including the desired component, at least one other component and water vapour, substantially as hereinbefore described with reference to Figs. 1A and 1B or Figs. 2A and 2B of the accompanying drawings.

11. Apparatus for use in the separation of

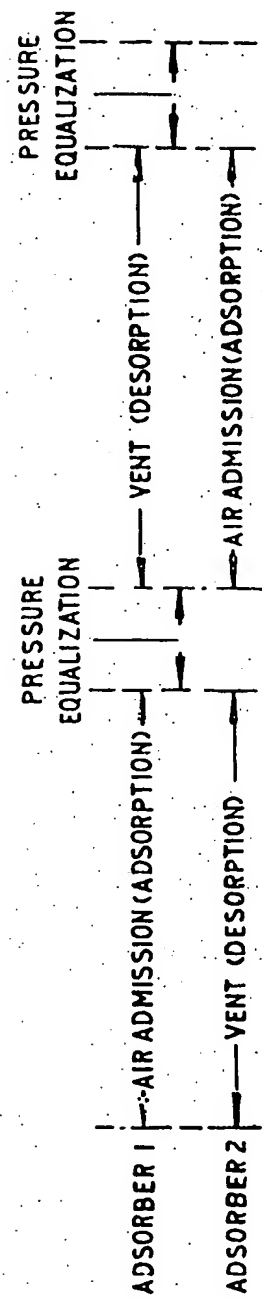
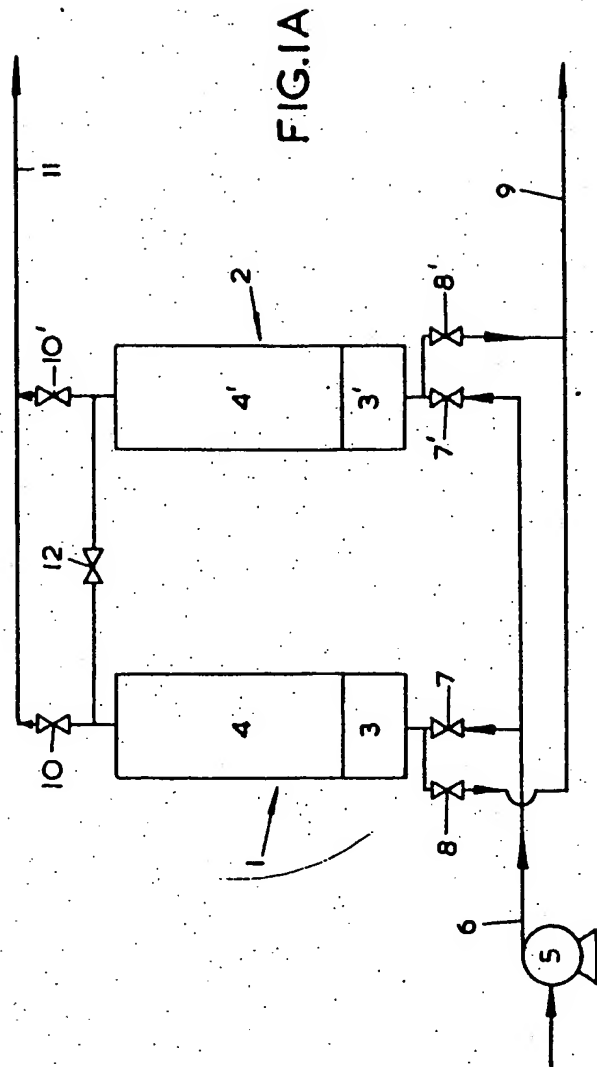
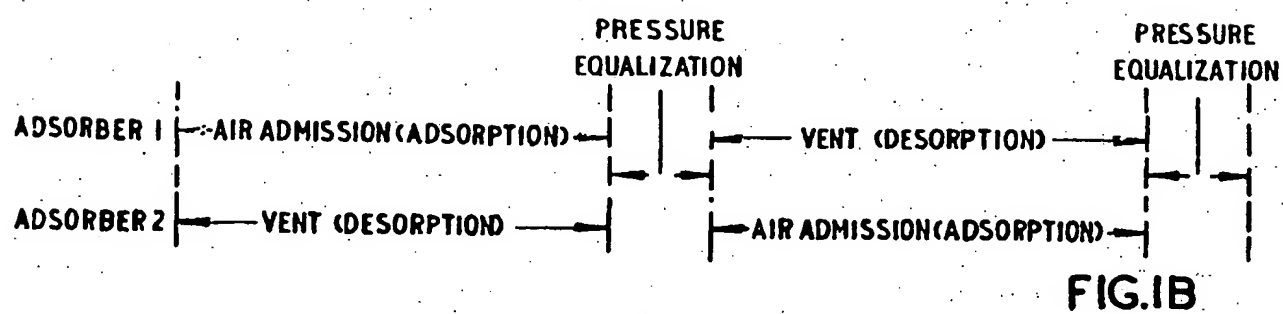
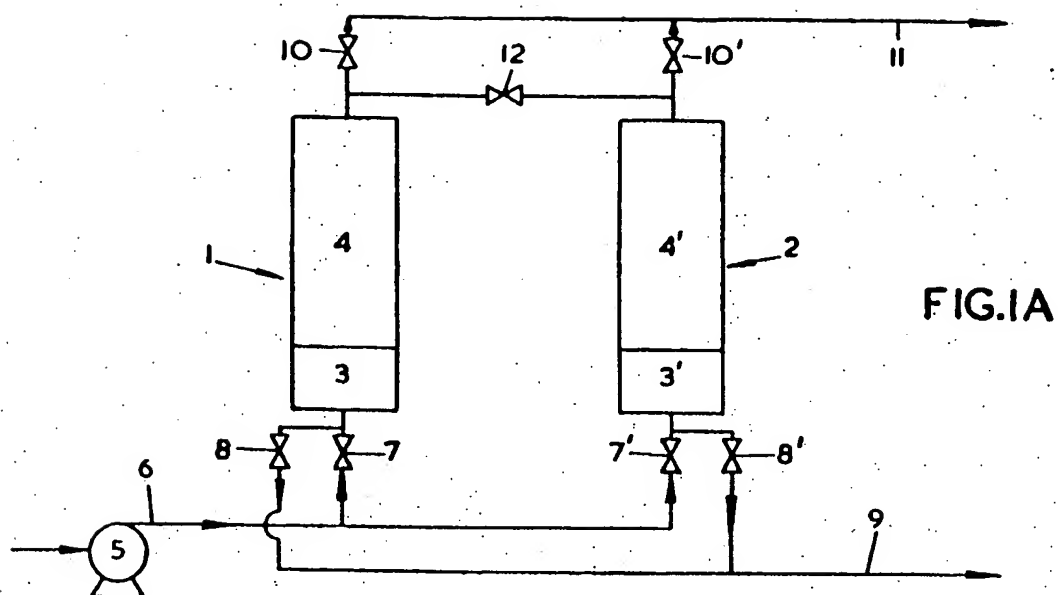
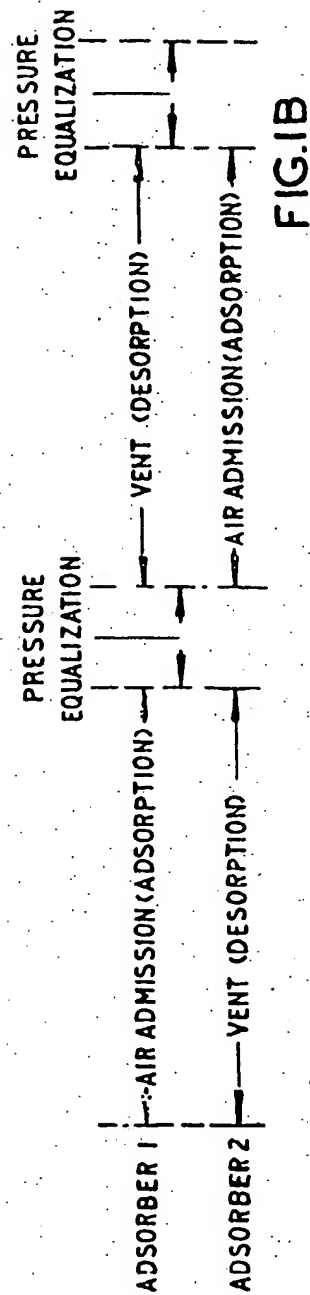
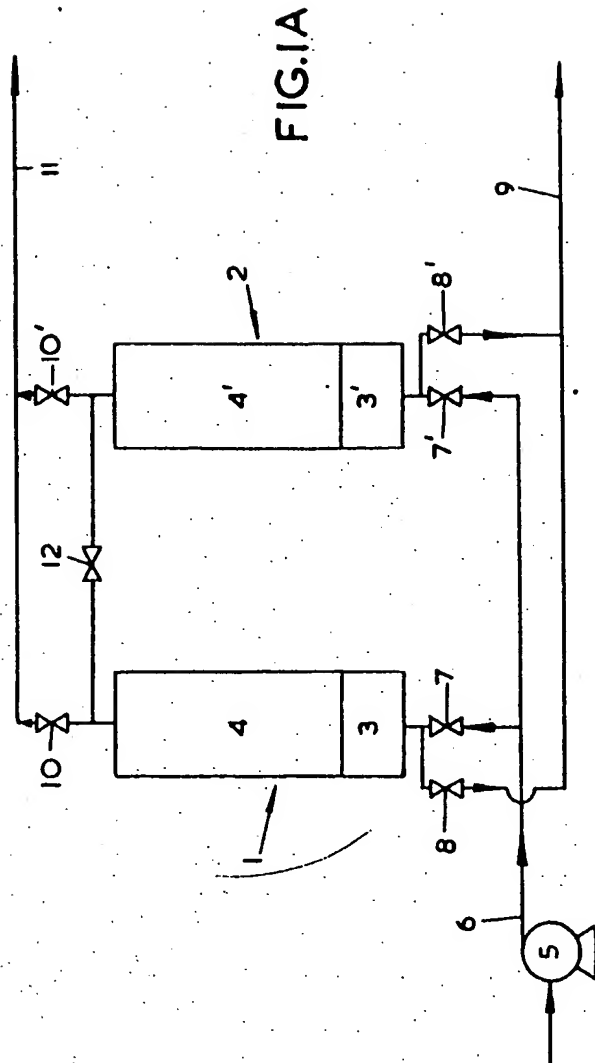
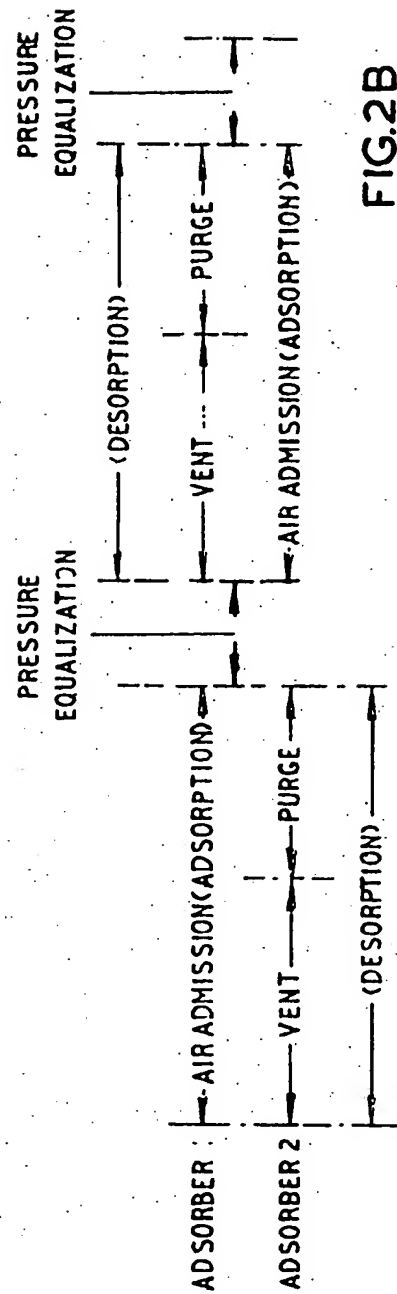
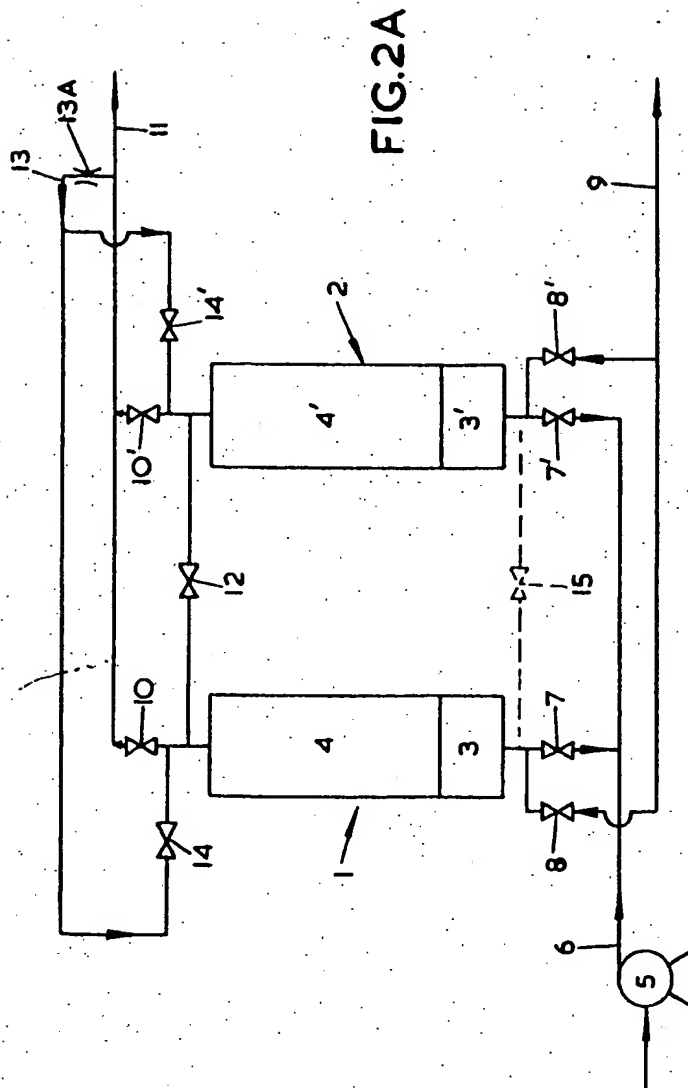


FIG. 1B










## Gas separation

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### Abstract

A pressure swing adsorption (PSA) process for the separation of a component from a gas mixture also containing water vapour, eg the separation of nitrogen from air, employs adsorbers 1 and 2 each having a first stage 3, 3' containing a desiccant adsorbent and a second stage 4, 4' containing an adsorbent which preferentially adsorbs another component of the mixture eg a carbon molecular sieve which adsorbs oxygen more rapidly than nitrogen. Each adsorber is put through a cycle comprising an adsorption step during which the mixture is passed through the adsorber by a compressor 5 and unadsorbed product is withdrawn through a delivery line 11, and a desorption step during which the adsorber is vented to atmosphere through a waste gas line 9. The pressure reached in the adsorber during such venting is the lowest reached during the entire cycle, no vacuum pump being employed. A valved line 12 allows pressure equalisation between the adsorbers. 

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